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ARMY RESEARCH LABORATORY

3-D Braided, Continuous Fiber Ceramic Composites Produced by Chemical Vapor Infiltration - SBIR

Mark D. Mello and Robert A. Florentine

ARL-CR-111 December 1993

prepared by

Quadrax Advanced Materials Systems, Inc.
300 High Point Avenue
Portsmouth, RI 02871

under contract

DAAL04-91-C-0004

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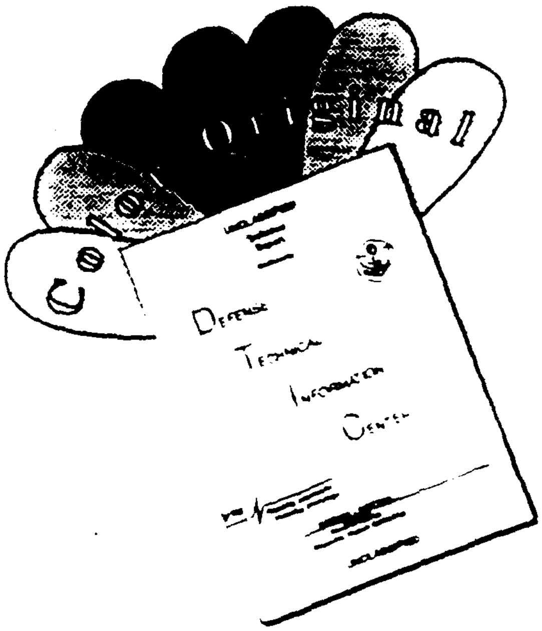

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REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*

2. REPORT DATE
December 1993

3. REPORT TYPE AND DATES COVERED
Final Report 1/18/91 - 1/2/92

4. TITLE AND SUBTITLE
3-D Braided, Continuous Fiber Ceramic Composites Produced by Chemical Vapor Infiltration - SBIR

5. FUNDING NUMBERS
Contract No. DAAL04-91-C-0004

6. AUTHOR(S)
Mark D. Mello and Robert A. Florentine

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Quadrax Advanced Materials Systems, Inc.
300 High Point Avenue
Portsmouth, RI 02871

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
U.S. Army Research Laboratory
Watertown, MA 02172-0001
ATTN: AMSRL-OP-PR-WT

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
ARL-CR-111

11. SUPPLEMENTARY NOTES
Michael J. Slavin COR

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; SBIR report, distribution unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

Continuous fiber reinforced ceramic composites have been successfully fabricated by chemical vapor infiltration of silicon carbide and silicon nitride matrix materials. Fiber preforms were three dimensionally braided with Nicalon™ and Nextel™ fiber materials forming a network of through thickness fiber architectures. Three unique material compositions were produced with the objective of demonstrating the capability of braiding brittle ceramic fibers and producing quality composites structurally capable of performing in a ballistic environment. It is anticipated that the continuous fiber architecture will be a significant technical advantage towards improvements in ceramic armor applications where non-catastrophic failure and increased toughness are a concern.

14. SUBJECT TERMS
Chemical vapor infiltration (CVI), Ceramic matrix composites, 3-D braid

15. NUMBER OF PAGES
30

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	UL

NSN 7540-01-280-5500

Standard Form 298 (Rev 2-89)
Prescribed by ANSI Std Z39-18
298-102

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ABSTRACT

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TECHNICAL OBJECTIVE

Ceramic materials have attractive properties that make their incorporation into various components and structures very desirable. This class of materials has high strength and thermal stability but are naturally brittle. Engineering approaches to this problem have developed various microstructural enhancements through solid phase toughening or whisker and particulate reinforcements.

The objective of the Phase I program is to demonstrate the ability of fabricating a continuous fiber reinforcement in a ceramic matrix. A three-dimensional (3-D) braiding technique was used to produce a network of fibers, or preform, to near net shape followed by infiltration of a ceramic matrix in between and around the fibers to form a composite material.

The fiber preform is produced to a specified architecture with controlled placement of fibers in a 3-D network. The fiber orientation is determined by the braiding angle and stepwise movement of fibers across the full dimension and thickness of the part. The composition of fibers throughout the matrix enhances the structural integrity of the component by minimizing the inherent brittle nature and low impact resistance of the ceramic matrix, thereby improving toughness and promoting non-catastrophic failure.

State of the art technology is available to process a host of ceramic oxide, carbide and nitride matrices for innovative compositions with continuous fiber reinforcements. Chemical vapor infiltration (CVI) was chosen to fabricate silicon carbide and silicon nitride matrices into Nextel™ and Nicalon™ fiber preforms in this program.

TECHNICAL BACKGROUND

A. 3-Dimensional Braiding Technology

The 3-D braiding technology, presently owned by Quadrax Advanced Materials, was developed and patented by Dr. Robert Florentine, formerly of Braidtech, Inc.[1] The original concept was designed as a technique to eliminate delamination as a failure mode in polymer matrix composites. Designated as Magnaweave, this technology has been evaluated and utilized by many prominent proponents in the academic and textile communities.

The braiding process basically manipulates fibers that are attached to elements assembled in a rectangular (or circular) grid with the opposite ends of the fiber gathered together some height above the grid. The rows and columns that make up the grid are free to move with the exception of the edge columns which are partially filled with elements. In operation, the following motion sequence is used repetitively:

1. All the rows are moved toward the empty spaces at the end of the rows as far as possible. One half of the rows move to the left and alternating rows move to the right.

2. All the columns are moved toward the empty spaces at the end of the columns as far as possible. One half of the columns move up and alternating columns move down.

3. Step 1) is repeated; Step 2) is repeated.

The motion sequence causes a single element to follow a diagonal path through the grid, resting at an edge before resuming its path. The motion of a single element is shown in Figure II.

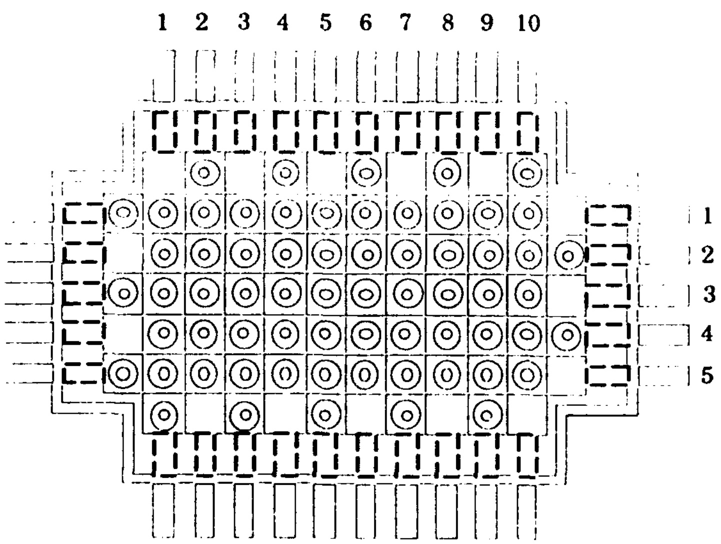

Figure I. MAGNAWEAVE Loom

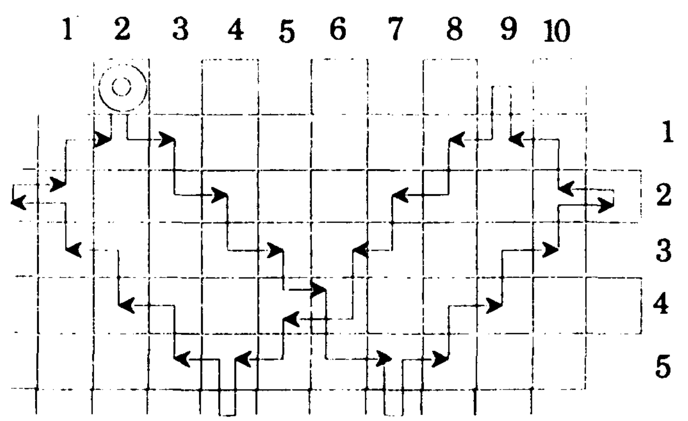

Figure II. Element Travel During Braidi

The braiding technology provides the ability to fabricate specific patterns and cell sizes. This flexibility is used to tailor fiber orientations to meet mechanical property requirements.

- The fiber placement determines the magnitude of the mechanical property in a given orientation due to the placement of the reinforcing fibers.

- The size and shape of the fiber cells and their interconnections dictate how, and to what extent, microcracks propagate. Preventing the growth of matrix cracks increases the overall properties and level of performance of the composite.

- Cell size and shape are a factor in the ease and completeness of the matrix infiltration process. Large cells (loose braid) may make matrix penetration easier, while tighter braid geometries may promote void content.

- Tight braid architectures may promote matrix retention under stress since the matrix should be more tightly anchored in the reinforcement network.

B. Chemical Vapor Infiltration

The CVI process is used to deposit material in the interstices of a fibrous preform. The material forms a matrix for a composite whose properties are enhanced by the presence of a fiber reinforcement.

As significant as the choice of processing parameters for the chemical vapor infiltration is for the consolidation of ceramic matrix composites, there is another aspect which is equally important - the choice of the architecture for the preform. Ceramic matrix composites have typically utilized non-continuous reinforcements. For some applications these are acceptable, but not for those requiring the highest of mechanical integrity.

It is difficult to penetrate the interstices of a preform by CVI without closing off the porosity at the surface and leaving voids near the center of the body. Gaseous diffusion of active precursors must be allowed to reach the inner-most surfaces of the preform without totally reacting on the outer fiber surfaces. This can be accomplished by controlling both the temperatures and pressures involved in the process. Deposition rates are slow and cause long processing times, typically (greater than 100 hours) in the case of carbon/carbon processing by CVI.

An alternate approach is to force the convection of reactive gases through the interstices by the maintenance of a pressure differential. This technique is regularly used at 3M Delta G for the infiltration of ceramic matrix composites. The result is an increase in the material deposition and a lower manufacturing cost.[2]

Woven preforms, 2-D lay-ups and true 3-D weaves, have been infiltrated by 3M Delta G. The 2-D materials have been generally unsatisfactory. Spontaneous delamination usually follows consolidation due to thermally induced strains from the mismatch in thermal expansion between the fibers and the matrix. 3-D woven structures have consolidated much better, but were previously expensive and difficult to make in a variety of shapes. Over the past two years, 3M Delta G has made more than 1000 CVI runs in connection with the development of 3M's SICONEX ceramic composites and have found braided preforms preferable to infiltrate than other architectures.[3]

TECHNICAL EFFORT

A. Program Requirements

Conversations with the project's technical monitor identified the major application interests as armor and gun barrel liners. For the purposes of specifying performance criteria, the application as an armor material was selected to define the braid architectures.

Typically, ceramic armor materials consist of a ceramic tile with a ductile backing material. Upon impact by a projectile, compressive loads are imposed which are translated into a compressive stress directed perpendicular to the armor face. If the compressive strength of the material exceeds the load, the stress travels through the armor until it strikes the rear surface. When the compressive force meets the interface between the armor and the backing material it is reflected back into the armor as a tensile stress.[4]

There are various methods of improving the fracture toughness of a ceramic material. Compositions of ceramic/ceramic and ceramic/metal materials can increase the strength and damage tolerance of the composite. The advantage of introducing a continuous fiber reinforcement is to increase the tolerance and to improve the fracture toughness and prevent catastrophic failure. Similar to micro-reinforcements, the continuous fiber architecture is intended to interfere and block propagating microcracks, preventing them from merging and

causing premature failure. Additionally, the fibers also provide directional reinforcement to greatly enhance the structural integrity by isolating damage in the fiber network.

Ceramic matrix processing techniques for fibrous preforms which are commonly available include:

- CVI
- polymer precursor pyrolysis
- slurry infiltration
- colloidal infiltration

Not all are commercially practiced and the availability of processing materials is limited. Polymer impregnation and CVI techniques have extensive experience processing continuous fiber preforms. CVI was selected for this program because of the high purity of the matrix material and the range of available materials for future investigation.

1. Candidate Fibers

The fiber materials selected for the technology demonstration are silicon carbide (Nicalon™) and alumino-silicate (Nextel™) because of their high strength, proven ease of handling and braiding, and commercial availability. The properties of the fibers are listed below.

<u>Nextel™ 312</u>

Tensile Strength	1.71 GPa	(250×10^3 psi)
Tensile Modulus	151.7 GPa	(22×10^6 psi)
Filament Diameter (900 denier)	10 - 12 μm	(.0004" - .0005")
Filament Density	>2.7 gm/cm^3	(> 168 lb/ft^3)
Chemical Composition	62% Al_2O_3, 24% SiO_2, 14% B_2O_3	

<u>Nicalon</u>™ - Ceramic Grade

Tensile Strength	2.8 GPa	(400×10^3 psi)
Tensile Modulus	193.1 GPa	(28×10^6 psi)
Filament Diameter (900 denier)	15 μm	(.0006")
Filament Density	2.5 - 2.65 gm/cm^3	(156.1 - 165.4 lb/ft^3)
Chemical Composition	ultra fine β-SiC crystals with excess carbon	

The objective of the program is to fabricate 3-D braided ceramic composites for mechanical testing. This achievement will suggest the implementation of a braided composite with the inherent advantages of a continuous fiber reinforcement to impart structural integrity to armor in multiple-hit situations.

B. Preform Geometry

The original solicitation requested coupon tiles of each composition to measure 50 x 50 x 10 mm for property evaluation. An alternative approach was considered. Rather than cut the ceramic tiles to the required thickness for mechanical test specimens, the strip preforms could be fabricated to the desired thickness. The advantages of this approach are multiple. Primarily, the test pieces will have a higher degree of fiber continuity throughout the piece. Cutting test pieces from a larger tile will result in the fibers having no length greater than the distance from one side of the test piece to the other. The continuous reinforcing characteristic of the fiber is diminished and the matrix strength then becomes increasingly important. Additionally, using preforms with the required thickness eliminates the need to cut pieces from large blocks. Such cutting procedures are expensive and may damage the test pieces themselves. The cutting process can generate incipient cracks in the matrix, providing a source for failure that seriously reduces the true capabilities of the composite. The technique employed produced narrow test strips with the same ease, quality, and cost as that involved in making thicker tile preforms.

C. Preform Fabrication

1. Selection of Fiber Architecture

Because of the exploratory nature of the project in its initial phase, one is inclined to design a braid architecture that will demonstrate maximized properties in one direction. Using the mechanical test results as a baseline, future architectures can be designed to meet specific properties.

Our selection for a demonstration preform architecture is based on maximizing the axial and through the thickness properties of the composite. To achieve that result, the selected braid architecture is defined by a 1 x 1 x 3 fiber orientation. Details of the loading scheme, fiber volume, and predictions of properties are based on this selection.

2. Preform Characteristics

Deliverable under the Phase I contract are nine tiles as follows:

2 coupons, 3mm x 50mm x 50mm of Nextel™ fiber/silicon carbide matrix
1 coupon, 10mm x 50mm x 50mm of Nextel™ fiber/silicon carbide matrix

2 coupons, 3mm x 50mm x 50mm of Nextel™ fiber/silicon nitride matrix
1 coupon, 10mm x 50mm x 50mm of Nextel™ fiber/silicon nitride matrix

2 coupons, 3mm x 50mm x 50mm of Nicalon™ fiber/silicon nitride matrix
1 coupon, 10mm x 50mm x 50mm of Nicalon™ fiber/silicon nitride matrix

The fibers employed are zero twist rovings. The fiber was purchased in 1,800 denier spools and four ends were plied with a light wrap of a PVA fiber. This retains the maximum properties of the fibers and provides a uniform fiber bundle for added ease during braiding.

The fiber volume of 0.3 - 0.4 was selected from previous experience with the CVI contractor. This is an acceptable range for insertion of matrix materials by vapor infiltration to guarantee uniform depositions.

The characteristics of the preforms are as follows:

	Set #1	Set #2	Set #3
1) Fiber	Nextel™ 312	Nextel™ 312	Nicalon™
2) Fiber Size (denier)	7,200	7,200	7,200
3) Fiber Volume	0.35	0.35	0.35
4) Fiber Treatment	PVA served	PVA served	PVA served
5) Fiber Area	$2.8x10^{-3}cm^2$	$2.8x10^{-3}cm^2$	$3.0x10^{-3}cm^2$
6) Matrix Composition	SiC	Si_3N_4	Si_3N_4

3. Loom Loading

The braider is loaded according to the braid geometry, the fiber dimension, the fiber volume and the dimensions and shape of the part to be braided. A manual braider was used for the demonstration purpose of this contract (Figure III).

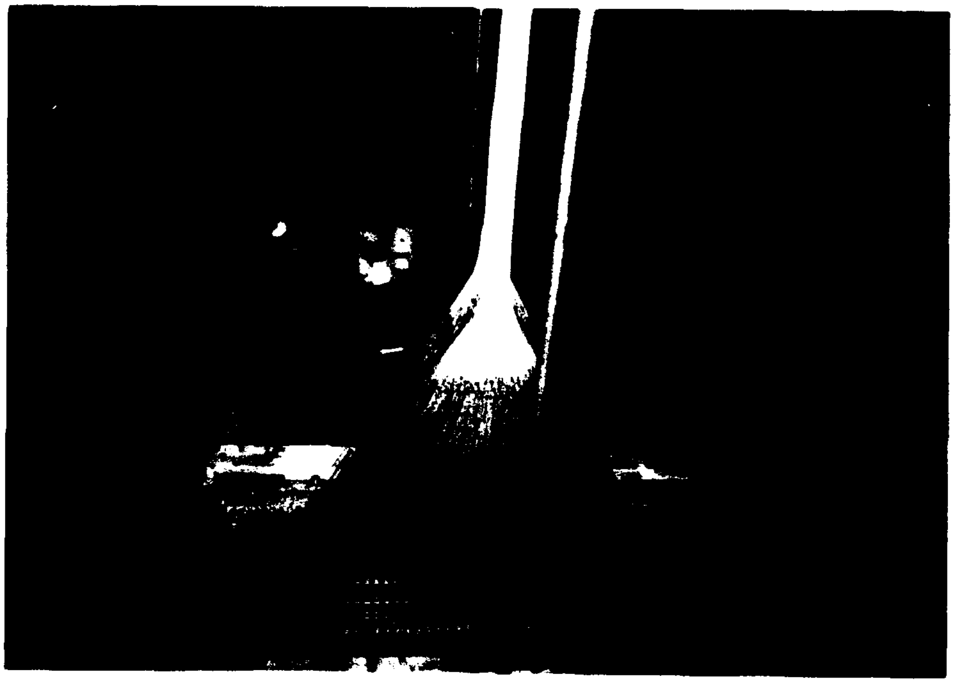

Figure III. Manual Loom Loading

This braider has twenty four rows and sixty elements per row. Each preform has its own loading pattern dictated by the thickness dimension and fiber type as listed below:

- Nicalon™ fiber - 10 mm thickness - 47 columns and 14 rows
- Nicalon™ fiber - 3 mm thickness - 47 columns and 5 rows
- Nextel™ fiber - 10 mm thickness - 41 columns and 14 rows
- Nextel™ fiber - 3 mm thickness - 41 columns and 5 rows

4. Loom Motion Sequences

The fiber strands were connected to elements in the loom. The rows and columns making up the loom were manipulated to produce the fiber preform. The machine motion was identical for each preform, so for brevity, only one example is presented.

Nextel™ Preform, 50 mm x 50 mm x 10 mm (Appendix A, Figure 1)

The active part of the loom is made up of 47 columns and 14 rows.

- rows #1 and #14 are fixed
- rows #2 - #13 are active
- rows #2,4,6,...12 are coupled to move as a unit
- rows #3,5,7,...13 are coupled to move as a unit
- columns #1 and #47 are fixed
- columns #2 - #46 are active

- rows #2 - #14 are loaded with elements
- columns #1,3,5,...45 are loaded with elements
- columns #2,4,6,...46 are loaded with elements

- coupled rows #3,5,7,...13 are positioned to the right
- coupled rows #2,4,6,...12 are positioned to the left
- columns #3,5,7,...45 are moved out away from the loom
- columns #2,4,6,...46 are moved in towards the loom

The first row motion

- move the odd numbered rows one space to the right
- move the even numbered rows one space to the left

The first column motion

- move the odd numbered columns one space in towards the loom
- move the even numbered columns one space out away from the loom

The braid point is positioned above the loom to produce a braid angle of 10∞.

The second row motion

- move the coupled sets one space in the opposite direction to the first row motion

The second column motion

- move each set of columns in the direction opposite to the first column motion

Comb the braid maintaining the required braid angle. Repeat each row and column sequence as above.

The thinner, 3mm Nicalon™ coupons were braided first. The additional fibers were added to the loom for the 10 mm thickness and the braiding was started at the end of the previous preform (Appendix A, Figure 2). The braid angle was maintained throughout, although it became difficult towards the end of the coupon length.

It was decided that a more efficient and easier method to fabricate the Nextel™ preforms was to braid them side by side at the same time. The loom loading pattern was set up to facilitate braiding both thicknesses with the same single motions while maintaining separate coupons. This method was very effective in producing quality preforms more easily and quickly.

RESULTS

A. Preform Quality

Sufficient lengths of braid were made, from which lengths slightly larger than 50 mm were cut to allow for subsequent handling and processing that might damage the loose fiber ends. The dimensions of the preforms were recorded and listed below.

Dimensions of Braided Preforms

Nextel™ - 3 mm braid

thickness	2.54 mm (.100")
width	50.8 mm (2.00")
surface angle	8 - 9°
total length	50.8 cm (20")

Nextel™ - 10 mm braid

thickness	10.46 mm (.412")
width	50.8 mm (2.00")
surface angle	8 - 10°
total length	50.8 cm (20")

Nicalon™ - 3 mm braid

thickness	3.56 mm (.140")
width	63.5 mm (2.5")
surface angle	8 - 10°
total length	19.69 cm (7.75")

Nicalon™ - 10 mm braid

thickness	12.07 mm (.460 "- 490")
width	60.45 mm (2.38")
surface angle	10°
total length	12.7 cm (5")

B. Composite Quality

An interface layer of pyrolytic carbon was deposited in the first step of chemical vapor infiltrating the fiber preforms. The benefits of such a coating are to increase the strength and fracture toughness of the composite by minimizing matrix adhesion to the fibers. The discrete boundary layer insures that fracture energy is not transferred directly from the matrix to the fiber thereby maintaining the integrity of the fiber as a structural reinforcement (results of characterization studies have been reported extensively in the literature [5,6,7]). Pyrolytic carbon was deposited by processing the preforms in propane gas at 1050° C for two hours. The

resulting interface coating was approximately 0.4 μm in thickness. Previous experience by 3M Delta G Corporation has indicated that this is an optimum thickness for enhancing the mechanical properties of 10-15 μm diameter fibers, such as those used in this program.

CVI of the matrix material followed, to form silicon carbide and silicon nitride matrices in the respective fiber preforms. The initial infiltration proceeded slowly while filling the gross voids between the fiber bundles. The process was completed at the point when the deposition was preferential as a surface coating rather than infiltrating the interstices of the preform. The quality of the final composite coupons is listed below:

Preform Fiber	Matrix Material	Thickness (mm)	Weight (gm)	Density (gm/cc)	Fiber Volume (%)	Matrix Volume (%)
Nextel	SiC	10	59.0	2.55	35	50
Nextel	SiC	3	20.5	2.55	35	50
Nextel	SiC	10	20.4	2.55	35	50
Nextel	Si_3N_4	10	50.1	2.16	35	45
Nextel	Si_3N_4	3	13.9	2.16	35	45
Nextel	Si_3N_4	3	14.8	2.16	35	45
Nicalon	Si_3N_4	10	67.2	1.98	30	45
Nicalon	Si_3N_4	3	20.6	1.98	30	45
Nicalon	Si_3N_4	3	25.4	1.98	30	45

Photographs of the ceramic composite coupons are in Appendix B.

The final densification to 75 - 85 % of total volume fraction was considered unacceptable for evaluation of mechanical properties. A parallel program ongoing between Quadrax and 3M Delta G had addressed a similar concern in attempting to achieve a high degree of densification. The approach chosen was to follow the CVI with a second process to increase the matrix volume in the composite. A polymer precursor of the matrix material was impregnated into the sample and pyrolized to form the ceramic material of interest. As a means of demonstrating this technique for the materials selected in this program, the Nextel™/silicon carbide coupons were chosen for further processing in this manner. To assist in optimizing the densification, the coupons were cut into the final dimensions for mechanical testing. These individual samples exposed more surface area, therefore allowing better impregnation of the precursor.

The additional processing increased the matrix volume by an additional 5 percent, bringing the total densification to 90 percent. The data are listed below:

Preform Fiber	Matrix Material	Thickness (mm)	Density (gm/cc)	Fiber Volume (%)	Matrix Volume (%)
Nextel	SiC	10	2.61	35	55
Nextel	SiC	3	2.73	35	55

CONCLUSION

Three ceramic composite materials were produced using a braided fiber preform and CVI matrix processing techniques. Although the degree of densification is not as high as expected and the mechanical data produced may not be representative of an optimized composite, an indication of the structural advantage of a braided, through thickness reinforcement, such as improved fracture toughness, will be demonstrated. Most importantly, the necessary improvements to achieve a much higher level of densification, as indicated in the Nextel™/silicon carbide samples, have been identified. As mentioned earlier, parallel efforts by the participating company have realized the necessity of supplying a preform with a higher fiber volume as a starting material, thus eliminating gross voids that are difficult to fill. Processing with a hybrid technique of CVI and polymer precursor impregnation additionally enhances the ability to achieve a high degree of densification. This learning experience occurred during this effort and therefore the deliverable samples did not benefit from the use of a higher fiber volume preform.

RECOMMENDATIONS

A Phase II proposal has been prepared for submission to the SBIR program. The proposal outlines a two year program to build upon the knowledge gained in the Phase I program, to develop the braiding technique to tailor the performance of continuously reinforced ceramic composites. The program has two phases: the first will fabricate various braided architectures and test their ballistic performance in a common matrix material; the second phase will fabricate the most promising fiber architecture in an engineered material system to demonstrate the effectiveness of the ceramic composite in ballistic and mechanical tests. The program will be

performed in cooperation with university and small business concerns in the field of ballistic protection.

A major effort of the program will include the research into state of the art matrix processing methods capable of producing fully dense composites in the materials of interest at a competitive price.

ACKNOWLEDGEMENT

Quadrax Advanced Materials Systems, Inc. recognizes the development opportunity presented by the U.S. Army's Small Business Innovation Research program. Specifically we appreciate the technical guidance from Michael J. Slavin at the U. S. Army Materials Technology Laboratory for the assistance in exploring our technology for application as armor materials. Acknowledgement for technical expertise in fabricating the materials in this contract is extended to Warren C. LaPointe of Quadrax and to 3M Delta G Corporation for their work on chemical vapor infiltration of the fiber preforms.

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APPENDIX A

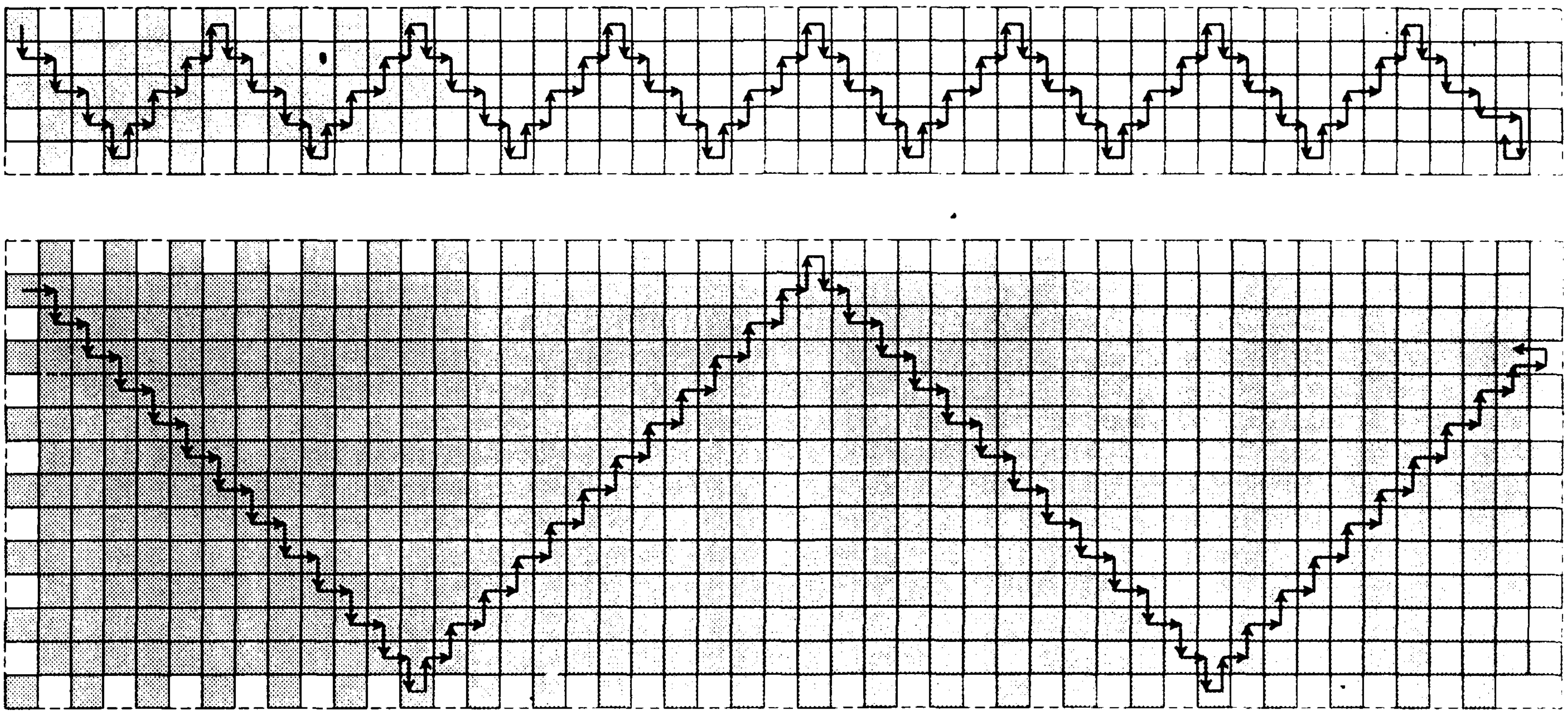

Figure 1. Nicalon Fiber Preform Motion Sequence

Shaded Areas Represent Loaded Elements

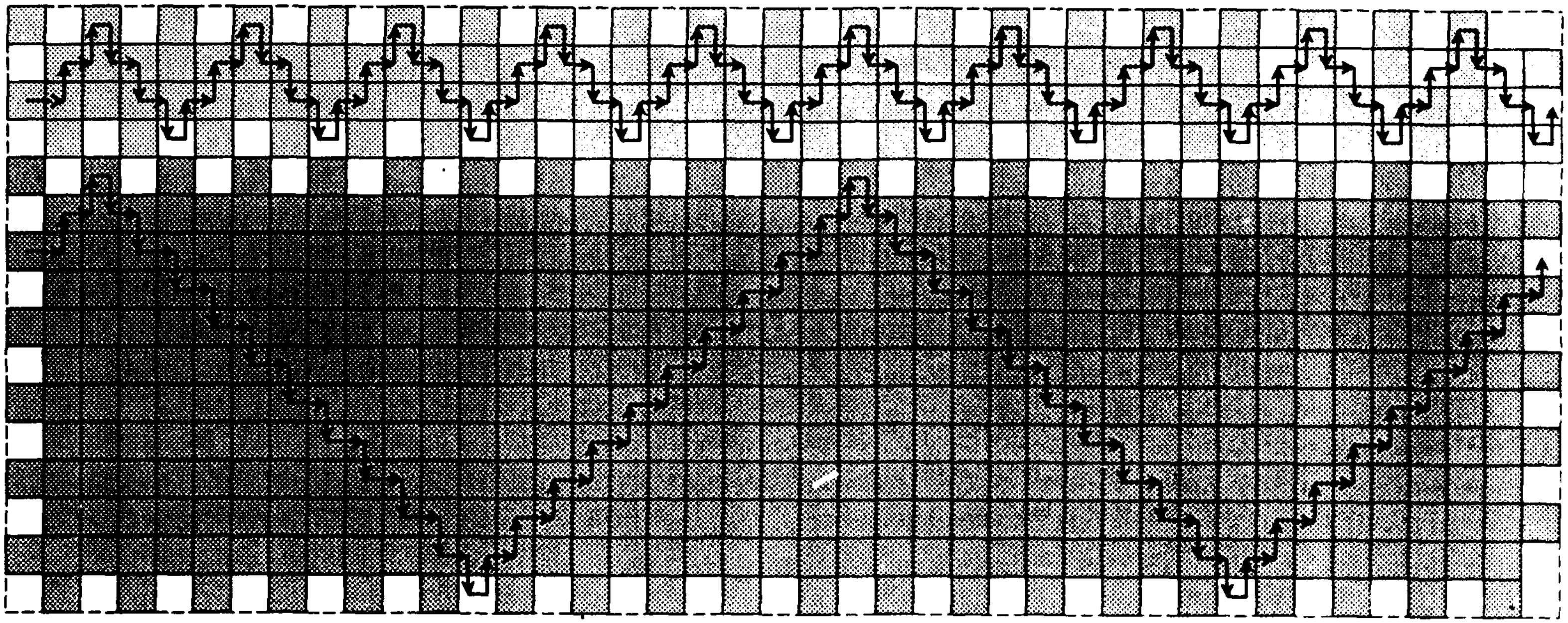

Figure 2. Nextel Fiber Preform Motion Sequence

Shaded Areas Represent Loaded Elements

APPENDIX B

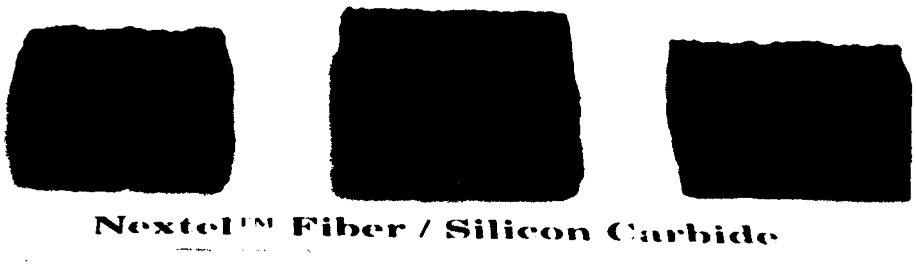

Figure 1A. Nextel™ Fiber/Silicon Carbide

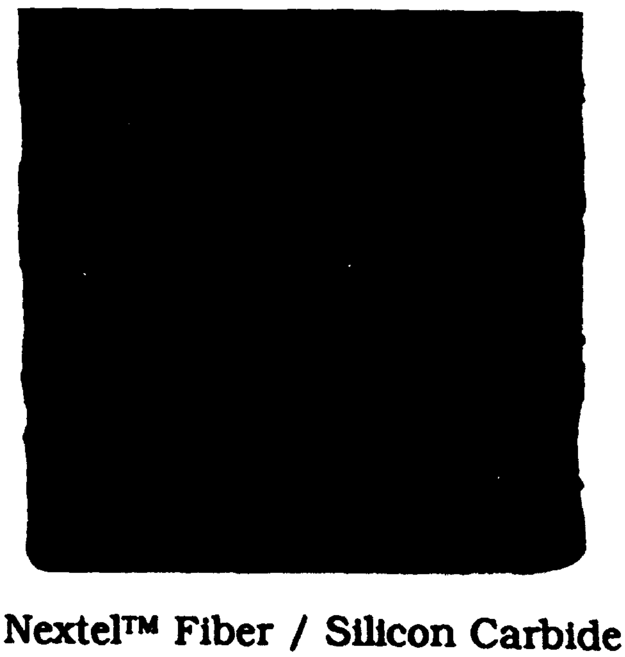

Figure 1B. Nextel™ Fiber/Silicon Carbide

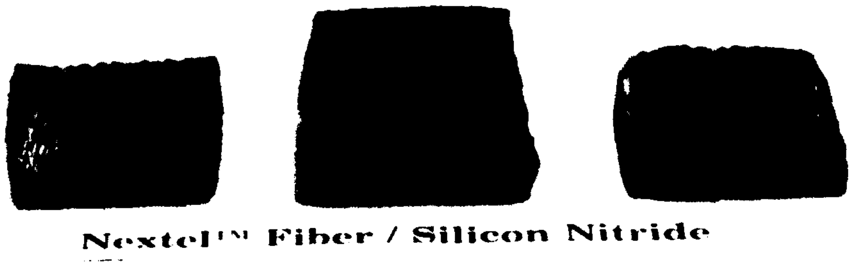

Figure 2A. Nextel™ Fiber/Silicon Nitride

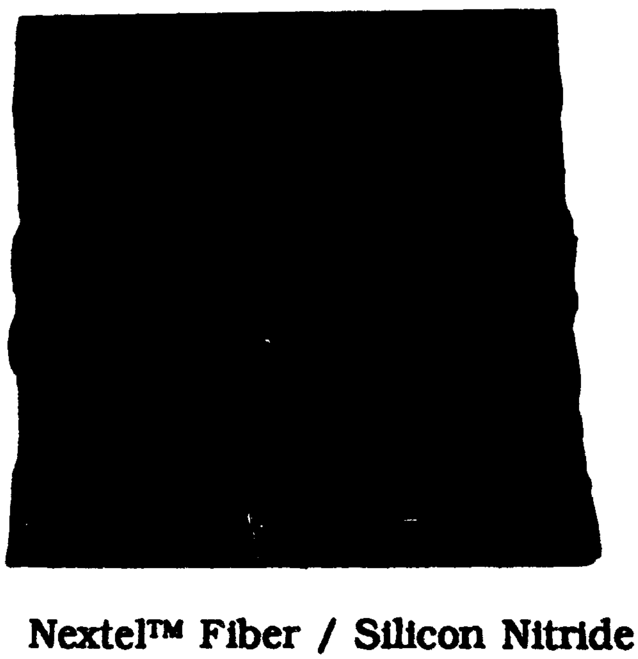

Figure 2B. Nextel™ Fiber/Silicon Nitride

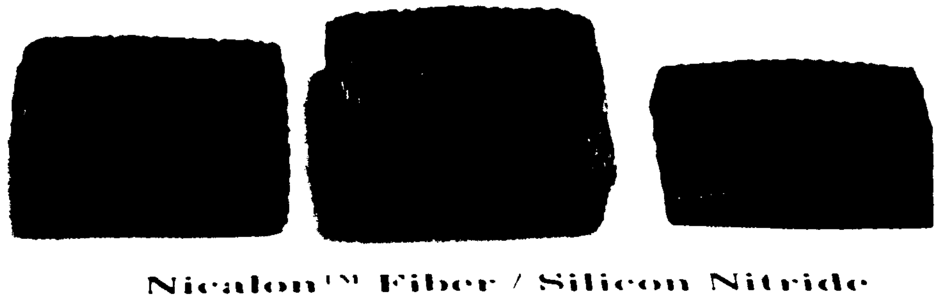

Figure 3A. Nicalon™ Fiber/Silicon Nitride

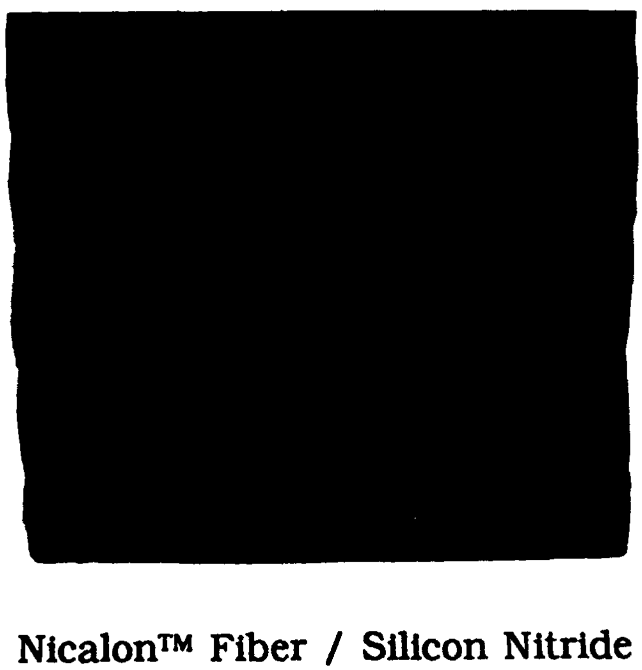

Figure 3B. Nicalon™ Fiber/Silicon Nitride

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